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Description

The invention relates to an apparatus for concentrating a suspension of crystals, consisting of a hollow cylindric column confining a concentration zone, the inner section of the column perpendicular to the centre line being constant, which column is provided at one end with means for the supply of suspension, with one or more filters near the other end of the column, with means for the removal of liquid passed through the filters with means for the removal of concentrated suspension at the other end of the column, and with means for the generation of a pulsating flow in the column.

The invention relates further to an apparatus for separating a mixture of substances by crystallization, characterised in that — seen in the direction in which the crystals move — a crystallizer is positioned upstream of a column confining a concentration zone as mentioned above.

Furthermore, the invention relates to a cascade for effecting a complete separation between crystals and melt by means of two or more of the apparatus mentioned above which are connected in series. However, a complete separation between crystals and mother liquor can only be obtained if the crystals are subjected to a counter-current wash process before leaving the last apparatus.

An apparatus as mentioned above is known from US patent specification 2,854,494. This patent specification describes a crystal wash column in which in a cylindrical room a crystal suspension is concentrated by directing this suspension along a portion of the cylindrical wall which contains one or more filters. At least a substantial portion of the liquid in the suspension is removed through these filters. The crystals substantially rid of liquid, thus the concentrated suspension, is transferred along the filters and removed by a way different from that of the liquid. Thus only part of the interior of the column forms a concentration zone. This zone ends beyond the filters 14. In this known apparatus the liquid and the crystals of the suspension to be treated initially move in the same direction. In the filters mounted in the wall, however, the greater part of the liquid is removed sideways, while the crystals continue to move in the same direction.

A serious disadvantage of this known apparatus is that a homogeneous flow of washing liquid through the crystal bed is not possible. Since the filters are exclusively located in the column wall, in the neighbourhood of the filters there is not an axial liquid flow which is equal over the entire cross section of the column, but a substantially radial liquid flow directed towards the filters. This causes a very undesirable preferential flow in the neighbourhood of the filters due to which the liquid does not flow to the filters along a short flow path but along a long one, part of which can be located — seen in the direction of the transport of the solid material — far beyond the filters. Needless to say that a uniform washing

out of the crystal bed over the entire cross section of the column and the obtaining of a homogeneous product become illusory. This disturbing effect of the filters mounted in the wall on the liquid flow in the crystal bed increases with a larger column diameter.

A second disadvantage of this known apparatus is that in the neighbourhood of the filter a densely packed crystal bed is formed, on which a big force has to be exercised for its transfer due to the fact that the friction between such a packed bed and the wall of the column can be very considerable. This big force exercised on the concentrated suspension involves a considerable energy consumption, while moreover there is the risk of deforming or damaging the crystals as a result of this big force. In the known apparatus according to the cited US patent specification the wall friction, and consequently also the force exercised on the concentrated crystal suspension, is decreased by maintaining a pulsating liquid flow in the column. Moreover, high demands are made upon the inside measurements of the columns, i.e. the inner cross section has to have the same size and shape all over the column in order to reduce the friction between the packed crystal bed and the wall to acceptable proportions. A narrowing of the inner cross section of the column would result in a compression in radial direction of the crystal bed and, consequently, the occurrence of excessively big friction forces. Due to a — gradual — widening of the inner cross section of the column in the direction of the material transfer the friction and the force to be exercised on the crystal bed could be reduced, it is true, but also an increasingly strong liquid flow between the wall and the crystal bed would occur disturbing a uniform liquid flow through the bed itself.

The object of the invention is to provide an apparatus for the concentration of a suspension which apparatus does not have the disadvantages mentioned above, or at least in a substantially reduced measure.

The invention is based on the insight that the liquid flow in a crystal bed in a column is disturbed substantially less when the filters are not, or at least not exclusively, mounted in the column wall, but evenly distributed over the entire cross section of the column. Such an array of the filters, however, must be such that the form and size of the cross section of the concentration zone are constant over the whole length of the column as in case of a decreasing cross section the concentrated crystal bed cannot be transferred, while in case of an increasing cross section the formation of channels occurs along the wall.

The object of the invention is, therefore, achieved with an apparatus as indicated above, which apparatus is characterized according to the invention in that one or more tubes of a constant outward diameter extend from the one end of the column in a direction parallel to the centre line through the column, and that in a portion of the wall of each tube at least one filter is mounted

3 forming the only direct connection between the interior of the tube and the interior of the column.

With this measure, naturally combined with a careful measurement of the interior of the column, on the one side a uniform removal of the liquid over the whole cross section of the column is obtained, and on the other side, also by the applied pulsating flow, an acceptable friction between the packed crystal bed, the wall of the column and the surface of the tubes is obtained.

The application of the apparatus according to the invention is not limited to suspensions of crystals but may also comprise suspensions of solid particles of another origin.

In a preferred embodiment of the apparatus according to the invention the column has a circular cross section, the tubes, if they are more than one, are distributed evenly over this cross section covering only a small portion of this cross section.

Preferably in each tube a portion of the wall with a length equal to a multiple of the outer tube diameter is carried out as a filter.

The filters can be mounted not only in the tube walls, but also in the wall of the concentration zone. Preferably all filters are equidistant from the end of the column.

The pulsating flow can be generated in many ways; in a preferred embodiment the rate of removal of the liquid passed through the filters is changed periodically, including the periodical closure of this removal. A pulsation device generally comprises a piston moving to and fro, and an expansion chamber in another part of the apparatus.

As no excessively big forces need to be exercised on the crystal bed in the device according to the invention the crystal bed is transferred exclusively by the pressure of the passing liquid. This prevents a.o. deformation and damage of the crystals in the bed. The concentration of the crystal bed increases in the direction of the filters and consequently also the pressure exercised by the liquid in axial direction of the bed. This pressure must be bigger than the opposing forces, mainly the friction. The force on the crystal bed is determined by the pressure drop of the liquid over the bed between the inlet of the suspension and the liquid removal through the filters. The pressure drop can be increased by extending the length of the bed or by increasing the flow rate of the liquid. If the suspension to be concentrated contains too little liquid to cause the desired pressure through the liquid on the crystal bed, a portion of the liquid passed through the filters can be recycled, as a result of which the pressure on the bed increases. In fact, this boils down to a decrease of the particle density of the suspension to be concentrated at the beginning of the concentration zone.

The concentrated suspension which has moved along the filters can e.g. be removed by means of a scraper.

The suspension concentrated in the apparatus according to the invention, which suspension has

4 been treated in this apparatus with a liquid in concurrent can then be treated in a washing zone in countercurrent with another liquid. This washing zone can form a unit with the apparatus according to the invention described above, and be mounted between the filters of the concentration zone and the means for the removal of the concentrated suspension. In the washing zone the concentrated suspension is transferred in the same direction in which it moved in the concentration zone, while at the end of the washing zone, averted from the concentration zone, a washing liquid is supplied which moves in countercurrent through the concentrated suspension. The washed concentrated suspension is preferably transported to a rinsing chamber where the concentrated and washed solid particles are again suspended in a liquid phase which is at the same time used as a washing liquid. The pulsating flow in the concentration zone can be generated by periodically changing the flow rate of the washing liquid to be supplied. When the apparatus according to the invention is used for the separation of a mixture of substances by means of crystallization, a crystallizer is — seen in the direction in which the crystals move — positioned before the concentration zone, in which crystallizer the mixture to be separated, which is liquid in normal conditions, is partly crystallized by cooling down, thus forming the crystal suspension which is supplied to the concentration zone. If the mixture of substances to be separated is solid in normal conditions, the crystal suspension can be prepared by partly melting this mixture. Further, a device can be mounted — seen in the direction of the movement of the crystals — after the washing zone, in which device the crystals or the crystal suspension removed from the washing zone, are molten and part of the melt is recycled as washing liquid to the washing zone.

The apparatus is provided with means for a controlled dosage of washing liquid. The control signal is obtained by measuring the temperature, concentration etc in the washing zone or in the rinsing chamber. The dosage of the washing liquid can be controlled by adjusting the pressure of washing liquid in the rinsing chamber.

If crystallization takes place from a melt as a result a so-called washing front occurs in the washing zone, i.e. the place where the temperature and the concentration gradients are maximal. The washing liquid supply is preferably controlled in such a way that this washing front is located in the washing zone about halfway the filters and the device for removal of the concentrated suspension.

Should the washing front be located at or in the neighbourhood of the filters, these could get clogged up by crystal growth.

In the case of crystallization from a solvent whereby the crystals are separated out as a solid product, a saturated solution of pure product in a solvent can be used as washing liquid. It is then advantageous to adjust the washing liquid supply in such a way that a small flow rate of washing

liquid flows through the entire washing zone and is removed through the filters.

In the case of crystallization from a solvent the apparatus can profitably be used as a preliminary stage for centrifuging or filtering. By connecting an apparatus according to the invention before a centrifuge or a filter a complete separation of mother liquor is obtained while a minimum quantity of washing liquid is used. The apparatus according to the invention can in particular be applied profitably for washing sugar crystals and anorganic salts such as cooling salt.

The crystals can move in the form a packed bed through the washing zone. It is recommendable that the filter tubes extend through the washing zone to the removal device, as otherwise in direct line with the tubes channels could be formed in the packed bed.

Another possibility is to mix the concentrated suspension and the washing liquid in radial direction so that there is no packed bed. Stirring can be effected by means of a stirring device comprising a rotor, the axis of which is coaxial with the centre line of the washing zone and which rotor is at least over part of its length provided with radially outwardly protruding rotor pins, while the washing zone is surrounded by a cylindrical mantle provided with radially inwardly protruding stator pins.

Furthermore, for the construction of this washing zone and the way in which the washing is done therein, reference is made to a second patent application filed in applicant's name on the same date as the present patent application [EP—A—83200919 (Priority: 22.06.82, Date of filing: 20.06.83)], which second patent application relates to a method and apparatus for treating suspended particles with a liquid in counter-current.

In the case of crystallization from the melt and heavily polluted mother liquors are used the desired complete separation between crystals and mother liquor cannot be realized by means of only an apparatus according to the invention with a packed crystal bed.

The crystallization temperature of such a mother liquor can be from 50° to 75°C lower than that of the pure main component to be separated. This means that under such circumstances the crystals in the column are able to be heated up by from 50° to 75°C. This is only possible if the heat required for this is withdrawn from the washing liquid. Due to this part of the washing liquid crystallizes which results in a decrease of the porosity of the packed bed. This decrease of the porosity may not be so strong that a total or at least substantial closing of the packed bed occurs. Since the specific heat of most substances amounts to about 1% of the melting heat the maximal allowable temperature difference is about from 20° to 40°C at a bed porosity of about from 0.4 to 0.5 which frequently occurs in a packed bed of crystals.

The required washing pressure for causing the flow rate of the washing liquid increases as the

bed porosity decreases. Due to this a greater driving force is needed for the forced crystal transfer against the washing pressure towards the melting circuit. In that case the crystal transfer consumes relative much energy and the hydraulic forces may be too small for realizing the desired capacity of the crystal transfer.

If there is a great temperature difference between the supplied crystals and the pure product a complete separation between crystals and mother liquor can nevertheless be realized if two or more apparatus according to the invention are connected to form a cascade. Often two apparatus are sufficient for bridging the temperature difference between the supplied suspension and the pure substance. The following observation is therefore and for the sake of simplicity limited to a cascade of two apparatus according to the invention.

The crystal suspension to be treated is supplied to the first apparatus and concentrated. The concentrated crystal mass is again suspended in the rinsing chamber with mother liquor from the second apparatus and is as diluted suspension transferred to the second apparatus.

The process conditions can be set in such a way that in each apparatus there occurs a stationary washing front. In the second apparatus a pure melt of crystals is again used as washing liquid. In the first apparatus the washing liquid is the mother liquor from the second apparatus.

The heat required for increasing the temperature of the crystals in the first apparatus is withdrawn from the mother liquor from the second apparatus which functions as washing liquid, due to which this mother liquor partially crystallizes.

Due to the partial crystallization of the mother liquor from the second apparatus which mother liquor functions as washing liquid, the concentration of the impurities increases and due to this the corresponding equilibrium temperature drops.

A controlled quantity of melt of pure crystals is fed to the rinsing chamber of the first apparatus in order to undo the change of the concentration caused by the extra crystal growth. Since the equilibrium temperature is a good measure for the composition of the liquid the addition of the melt to the rinsing chamber is controlled based on a temperature measurement in the rinsing chamber.

Preferably a cascade of two apparatus according to the invention is provided with means for a sluice in the pipe line between the mother liquor removal in the second apparatus and the inlet of the rinsing chamber in the first apparatus.

By using a sluice a complete separation of crystals and mother liquor is possible in such a cascade without the occurrence of a stationary washing front in the first apparatus.

When a sluice is used a small quantity of mother liquor together with the concentrated crystal mass is removed in the first apparatus towards the rinsing chamber. During the passage

of the crystals through the first apparatus the crystals remain in contact with mother liquor having the original composition. Thus it is possible to separate a substantial part of the mother liquor at a constant temperature of the crystals. The required heat is withdrawn in the rinsing chamber from the liquid phase. The extra crystal growth takes place in a diluted suspension. In this way any temperature difference can be bridged since no closing and clogging of the crystal bed occurs.

The liquid which is removed through the sluice is recycled to the crystal bed. The quantity that is sluiced is not only determined by the quantity of mother liquor that is allowed to be carried along together with the crystals in the first apparatus, but also by the concentration difference between the mother liquor and the liquid in the rinsing chamber (which is the mother liquor of the second apparatus). As the impurity concentration is higher in the rinsing chamber, the mother liquor carried along is less diluted and a smaller quantity is sluiced which results in a smaller loss factor due to the sluice.

Instead of pure product the starting material to be crystallized can often be fed to the rinsing chamber of the first apparatus in order to undo the change of the concentration caused by the extra crystal growth. The addition of feed to the rinsing chamber of the first apparatus has the advantage of heat recuperation as the cold content of the crystals transferred by the first apparatus can be used for partial crystallization of the starting material.

The invention will be explained with reference to the drawing.

Fig. 1 shows a diagram of the apparatus according to the invention for concentrating a suspension of crystals.

Fig. 2 is a diagram of the apparatus of fig. 1 combined with a washing zone which is stirred.

Fig. 3 is a diagram of a cascade of two apparatus connected in series each with a zone with a packed bed. Pure product is fed to the rinsing chamber of the first apparatus, from which product the heat can be withdrawn required for increasing the temperature of the crystals.

Fig. 4 is a diagram of a cascade of two apparatus according to the invention connected in series, in which, instead of pure product the starting material to be crystallized is fed to the rinsing chamber of the first apparatus.

The device according to fig. 1 consists of a hollow cylindrical body 1 of e.g. glass, with one or more tubes 2 in it, positioned in longitudinal direction and each provided with a filter 3 over a length which is a multiple of the diameter of the tube. The tubes are mounted in a fixed place in the column and evenly distributed over its inner cross section. The cross section of the tubes covers only a fraction of the surface of the inner cross section of the cylinder. At one end the apparatus is provided with means for the removal of concentrated suspension, viz. a rotatable knife

14 with a drive 22. At the other end a line 5 is available for supply of suspension, which line 5 ends in a mixing chamber 15 from which the suspension is carried into cylinder 1, evenly distributed over its cross section. The filtered liquid is removed through lines 6 and a pump 7.

The filters are located in such a way that the distance between the suspension inlet and the filters is three to four times the distance between the suspension outlet and the filters.

The wall friction at the filters 3 can be reduced by generating a pulsating liquid flow in the removal line of the liquid by means of a piston in a cylinder 8.

The suspension to be concentrated is a crystal suspension, which can be prepared in a crystallization vessel 9 with a scraped cooler 10. The desired mixing in vessel 9 is obtained by a stirrer 11. The mixture to be crystallized is supplied through a line 12; the mother liquor can be removed through a control valve 13 and/or be recycled by a pump 7 as the circumstances may require.

Fig. 2 is a diagram of a combination of the apparatus of fig. 1 with a washing zone which is stirred.

The reference number 1 substantially indicates the apparatus according to fig. 1. Corresponding component parts are indicated with the same reference numbers as in fig. 1.

Between the filters 3 and the crystal removal 30 a stirring device is mounted for tangential stirring the concentrated crystal suspension. This stirring device consists of a rotatable axis 23 with rotor pins 24, in combination with stator pins 25 fixed in the wall. The rotatable axis 23 is in direct line with the central filter pipe 31 in order to hamper the transfer of the concentrated crystal suspension as little as possible.

The crystal suspension is then transferred via a rotatable knife or scraper 14 with a drive 22 to a melting circuit consisting of a rinsing chamber 27, a heat exchanger 28 and a pump 29. A substantial portion of the crystals is removed as product through a control member 32. The other part of the molten crystals is recycled as washing liquid to the washing zone.

A portion of the washing liquid crystallizes in the washing zone and consequently the volume fraction of the solid matter increases in the washing zone. By using a tangential stirring, however, it can be provided that the crystals remain loose in respect of each other and that the washing pressure does not increase. By mounting a dynamometer 21 between the drive of the stirring device 20 and the rotatable axis 23 the torque exercised on the stirrer can be measured and on the basis thereof the volume fraction of solid matter in the crystal mass in the washing zone can be controlled.

The wall friction is reduced by a pulsating liquid flow caused by a piston 33 at the removal of the crystals.

Figure 3 is a diagram of two apparatus according to the invention connected in series. Both

apparatus consist of a washing zone with a packed crystal bed. In the first apparatus to which the suspension is fed corresponding component parts are indicating the same reference numbers as used in figures 1 and 2, but increased by 100. In the second apparatus the reference numbers of figures 1 and 2 are increased by 200. The number of filter pipes in each apparatus amounts to 10—100 per square meter of cross section of the column. A crystal suspension from a crystallizer is fed to apparatus 101 via line 141 and by means of pump 140. Via line 142 the mother liquor is recycled to the crystallizer and if needed partially sluiced as a waste flow. The concentrated crystal mass obtained in 101 is via a rotatable knife or scraper 14 transferred to rinsing chamber 127 and suspended with mother liquor from the second apparatus 201 which is fed to the rinsing chamber 127 through line 242. The crystals from 101 are suspended in this mother liquor and fed to the second apparatus by means of pump 240. In the second apparatus the suspension is again concentrated, the crystals are transferred to the rinsing chamber 227 by means of the knife 214 and thereafter they are suspended, molten by means of heat exchanger 228 and substantially removed as molten product by control means 232. A complete separation between crystals and mother liquor is effected by establishing a stationary washing front by means of a temperature sensor 234.

By means of a control means 148 such a value of the sluice is set (to be read by means of flow meter 147) that in this apparatus 101 no stationary washing front occurs in the packed bed, but that a minimal quantity of mother liquor is removed together with the crystal mass. Thus the crystals remain in contact with the original mother liquor and only just under the knife there is a limited temperature rise of the crystals. In the rinsing chamber 127 the crystals are suspended with mother liquor from 201 having a lower impurity concentration and consequently a higher equilibrium temperature. Thus the temperature of the crystals increases. The heat required for this is withdrawn from the liquid phase which due to this partially crystallizes.

The supply of a melt of pure crystals by means of control means 146 and temperature sensor 145 sets the temperature of the mother liquor and thus also the impurity concentration in the rinsing chamber at a certain desired value.

The liquid pressure level of the apparatus 201 can relative to the liquid pressure level of the apparatus 101 be increased according to the needs by means of throttle valve 249. By controlling the throttle valve 249 by pressure sensor 250 one is assured that possible washing pressure changes in the rinsing chamber 127 do not influence the liquid pressure level in the apparatus 201.

The mother liquor which together with the crystals is removed in the apparatus 101 to the rinsing chamber 127 is diluted with the melt supplied via 146 until the set value is obtained.

The sluiced material is removed to a suitable point in the crystallization cascade.

In order to dilute as little as possible the mother liquor which together with the crystals is removed in the apparatus 101 to the rinsing chamber 127, the impurity percentage of the mother liquor circulating between the apparatus 101 and 102 is preferably chosen as high as is still compatible with a good functioning of the apparatus 201.

Possible fluctuations in the crystal transfer are compensated by controlling the liquid flow rate through the crystal bed based upon a partial recycling of the mother liquor in such a way that the position of the crystal front, i.e. the position of the transition from diluted suspension to the compact bed is kept stationary. Keeping the crystal front at the same place in the apparatus 101 and 102 is realized by means of sensors 143 and 243 for measuring the difference between the dragging forces of the suspension and the bed and the pumps 144 and 244.

It is clear that in this way with a cascade of two or more apparatus according to the invention any occurring temperature difference between fed crystals and crystals in contact with a pure melt can be bridged.

Figure 4 shows a cascade of two apparatus according to the invention connected in series, in which no melt of pure crystals is supplied to the rinsing chamber of the first apparatus, but part of the starting material to be crystallized. Corresponding component parts are indicated with the same reference numbers as in figure 3. The only difference with the diagram of figure 3 is, that instead of melt of pure crystals starting material to be crystallized is fed by means of pumps 149 to rinsing chamber 127, and that the sluice is controlled on the basis of a temperature measurement in the rinsing chamber.

The heat required for increasing the temperature of the crystals transferred by 101 is withdrawn from the supplied starting material which due to this partially crystallizes.

When the starting material can already partially crystallize in the rinsing chamber 127 less heat has to be withdrawn in the crystallizer. Thus the heat exchanging surface in the crystallizer can be smaller. The supply of starting material to the rinsing chambers of the first apparatus consequently does not only lead to energy saving but also to a decrease of the investment cost.

If the composition of the mother liquor circulating between 101 and 201 can be chosen in such a way that the impurity percentage is some units lower than that of the starting material even the total feed can be supplied to the rinsing chamber 117.

Example I

The purification of para-xylene was carried out in a separation apparatus which substantially corresponded to the diagram of figure 2.

The crystallizer consisted of a simple outwardly scraped and inwardly cooled heat exchanger with a length of 500 mm and a diameter of 300 mm

positioned in a vessel with a length of 600 mm and a diameter of 500 mm. The heat exchanger and the vessel were substantially made of stainless steel.

The suspension concentrator had a length of 1000 mm and an inner diameter of 200 mm and contained seven filter pipes, each with a diameter of 25 mm. The length of the filters was 40 mm, the diameter of the filter openings 0,5 mm, and the filters were mounted at 300 mm from the crystal removal side.

Between the filters and the scraping knife a rotatable axis with rotor pins was mounted. In seven layers perpendicular to the axis rotor pins were mounted with a diameter of 8 mm; each layer comprised eight pins. The distance between the layers was 25 mm. As many stator pins 5 were mounted in the wall. The frequency of the liquid pulse was 1 sec.⁻¹ and the pulse mass about 0.2 kg.

The feed mixture consisted of para-xylene polluted with 20 wt.% ortho-xylene, meta-xylene and ethylbenzene. The temperature in the crystallizer was -20°C. Of the total feed of 270 kg/h 180 kg were crystallized, while the 90 kg of non-crystallized liquid was removed as waste flow.

The waste flow contained still about 40% para-xylene. At the side of the crystal removal 240 kg of crystals per hour were molten and about 180 kg/h were removed as 99.97% pure product (para-xylene).

The circulation rate of the mother liquor amounted to 1500 l/h.

A production of 180 kg/h yields at 8000 working hours per year an annual production of 1440 tons. With larger apparatus of the present type capacities of 100,000 tons and more per year can be realized.

Example II

The separation of para-xylene crystals from mother liquor was carried out in a separation apparatus which substantially corresponded with the diagram of figure 3.

The apparatus 101 and 201 had both a length of 1000 mm and an inner diameter of 150 mm and each contained six filter pipes with a diameter of 20 mm. The length of the filters amounted to 30 mm, the diameter of the filter openings to 0.3 mm and the filters were situated at a distance of 300 mm from the crystal removal side.

By means of pump 140 a mixture was supplied containing 100 kg/h of para-xylene crystals and 500 kg/h of mother liquor comprising about 70% wt. of impurities. The temperature of this mixture amounted to -30°C. The temperature of the rinsing chamber was kept at -10°C. Through 146:6 kg/h of a melt of pure crystals was supplied. Through 148 1.6 kg/h liquid containing 55% wt. of para-xylene was removed. The circulation rate of 240 amounted to about 500 kg/h. Through 232 100 kg/h of 99.97% wt. pure para-xylene could be removed.

A production of 100 kg/h yields at 8000 working hours per year an annual production of 800 tons.

With larger apparatus of the present type capacities of 100,000 tons and more per annum can be realized.

5 Claims

1. An apparatus for concentrating a suspension of crystals, comprising a hollow cylindrical column (1) confining a concentration zone, the inner section of the column (1) perpendicular to the centre line being constant, which column (1) is provided at one end with means for the supply of suspension (5, 15), with one or more filters (3) near the other end of the column (1), with means for the removal of liquid (6) passed through the filters (3), with means for the removal of concentrated suspension (14, 30) at the other end of the column (1), and with means for the generation of a pulsating liquid flow (8, 33) in the column (1), characterized in that one or more tubes (2) of a constant external diameter extend from the one end of the column (1) in a direction parallel to the centre line through the column (1), and that in the wall of each tube (2) at least one said filter (3) is mounted forming the only direct connection between the interior of the tube (2) and the interior of the column (1).

2. An apparatus according to claim 1, characterized in that the column (1) has a circular cross section and that if the tubes (2) are more than one they are distributed evenly over the cross section covering only a small portion of this cross section.

3. An apparatus according to one or more of the preceding claims, characterized in that at each tube (2) a portion of its wall with a length equal to a multiple of the outer tube diameter is carried out as a filter (3).

4. An apparatus according to one or more of the preceding claims, characterized in that the means for generating a pulsating flow in the column (1) consist of a piston (8) which periodically changes the rate of the removal of the liquid passed through the filters (3).

5. An apparatus according to one or more of the preceding claims, characterized in that means (5, 7, 9) are available for recycling part of the liquid passed through the filters (3) to the one end of the column (1).

6. An apparatus according to one or more of the preceding claims, characterized in that the means (30) for the removal of concentrated suspension at the other end of the column (1) comprise a scraper (14).

7. An apparatus according to one or more of the preceding claims, characterized in that between the filters (3) of the column (1) and the means for the removal of concentrated suspension (14, 30) a washing zone is provided with a supply pump (29) for washing liquid.

8. An apparatus according to claim 7, characterized in that the means for the removal of the concentrated suspension (14, 30) debouches into a rinsing chamber (27) and that means are provided for re-suspending the concentrated suspension.

9. An apparatus according to one or more of the preceding claims, characterized by means for a controlled dosage of washing liquid (29, 32).

10. An apparatus according to one or more of claims 7—9, characterized in that in the washing zone a stirring means (23, 24, 25) is provided consisting of a rotor (23, 24) the axis (23) of which is coaxial with the centre line of the washing zone, which rotor (23, 24) is provided, at least over part of its length, with radially outwardly protruding pins (24) and that the washing zone is surrounded by a cylindrical mantle provided with radially inwardly protruding pins (25).

11. An apparatus according to one or more of claims 7—10, possibly in combination with one or more of the claims 1—3 and 5 and 6, characterized in that the means for generating a pulsating flow in the concentration zone comprise a piston (33) which periodically changes the flow rate of the washing liquid to be supplied.

12. An apparatus for separating a mixture of substances by crystallization, characterized in that — seen in the direction in which the crystals move — a crystallizer (9) is positioned before the means (5, 15) for the supply of suspension to a column according to one or more of the preceding claims.

13. An apparatus for separating a mixture of substances by crystallization consisting of an apparatus for concentrating a suspension according to one or more of claims 7—11 and according to claim 12, characterized in that — seen in the direction of the crystal flow — after the means (30) for the removal of concentrated and washed crystal suspension a device (28, 29, 32) is positioned for completely melting and subsequently partly recycling as washing liquid to the washing zone the removed concentrated and washed crystal suspension.

14. Cascade for separating crystals from a suspension comprising a melt, characterized in that the cascade consists of at least two apparatus (101, 201) according to one of claims 1—11, or claim 13, which are connected in series, the first apparatus of the series also being according to claim 12, that the supply side of the column (101) of the first apparatus is provided with a supply line (141) for suspension, that the outlet (142) of the filters (103) of the first apparatus (101) is provided with a recycle line (144) for mother liquor, that the outlet (240) of the rinsing chamber (127) of the first apparatus (101) is connected with the supply side of the concentration zone of the second apparatus (201), that the outlet of the filters (203) in the second apparatus (201) is connected with the supply (242) of the rinsing chamber (127) of the first apparatus (101) and that the rinsing chamber (227) of the second apparatus (201) is connected through a heat exchanger (228) with a recycle line to the rinsing chamber (127) of the first apparatus (101) and a product outlet line respectively.

15. Cascade for separating crystals from a suspension comprising a melt according to claim 14, characterized in that the conduct (242)

between the outlet (202) of the filter (203) in the second apparatus (201) and the supply of the rinsing chamber (127) of the first apparatus (101) is provided with a sluice (148).

16. Cascade for separating crystals from a suspension comprising a melt according to claim 15, characterized in that the rinsing chamber (127) of the first apparatus (101) is provided with a supply (149) for fresh feed and that the sluice (148) is controlled (145) by the temperature in the first rinsing chamber (127).

17. Cascade for separating crystals from a suspension comprising a melt according to claims 14, 15 or 16, characterized in that the cascade consists of more than two apparatus connected in series.

Patentansprüche

1. Vorrichtung zum Konzentrieren einer Suspension von Kristallen, die eine hohle, zylindrische Kolonne (1) aufweist, die eine Konzentrationszone begrenzt, wobei der innere Teil der Kolonne (1) senkrecht zur Mittellinie konstant ist, bei der die Kolonne (1) an einem Ende mit einer Einrichtung (5, 15) zum Zuführen der Suspension, mit ein oder mehreren Filtern (3) in der Nähe des anderen Endes der Kolonne (1), mit einer Einrichtung zum Abziehen der Flüssigkeit (6), die die Filter (3) passiert hat, eine Einrichtung (14, 30) zum Abführen der konzentrierten Suspension am anderen Ende der Kolonne (1) und mit einer Einrichtung (8, 33) zum Erzeugen eines pulsierenden Flüssigkeitsstromes in der Kolonne (1) versehen ist, dadurch gekennzeichnet, daß ein oder mehrere Rohre (2) von einem konstanten Außendurchmesser sich von einem Ende der Kolonne (1) in eine Richtung parallel zur Mittellinie durch die Kolonne (1) erstreckt, und daß in der Wand jedes Rohres (2) wenigstens ein Filter (3) angebracht ist, der die einzige direkte Verbindung zwischen dem Innern des Rohres (2) und dem Innern der Kolonne (1) bildet.

2. Vorrichtung nach Anspruch 1, dadurch gekennzeichnet, daß die Kolonne (1) einen Kreisquerschnitt hat, und daß, wenn mehr als ein Rohr (2) vorgesehen ist, diese gleichmäßig über den Querschnitt verteilt sind und nur einen kleinen Teil des Querschnitts einnehmen.

3. Vorrichtung nach einem oder mehreren der vorangehenden Ansprüche, dadurch gekennzeichnet, daß an jedem Rohr (2) ein Teil seiner Wand mit einer Länge, die gleich einem Mehrfachen des Rohraußendurchmessers ist, als ein Filter (3) ausgeführt ist.

4. Vorrichtung nach einem oder mehreren der vorangehenden Ansprüche, dadurch gekennzeichnet, daß die Einrichtung zum Erzeugen eines pulsierenden Stromes in der Kolonne (1) aus einem Kolben (8) besteht, der periodisch die Abzugsrate der die Filter (3) passierenden Flüssigkeit ändert.

5. Vorrichtung nach einem oder mehreren der vorangehenden Ansprüche, dadurch gekennzeichnet, daß Einrichtungen (5, 7, 9) vorhanden

sind, die einen Teil der durch die Filter (3) gegangenen Flüssigkeit zu einem Ende der Kolonne (1) zurückführen.

6. Vorrichtungen nach einem oder mehreren der vorangehenden Ansprüche, dadurch gekennzeichnet, daß die Einrichtung (30) zum Abführen der konzentrierten Suspension am anderen Ende der Kolonne (1) einen Abstreifer (14) aufweist.

7. Vorrichtung nach einem oder mehreren der vorangehenden Ansprüche, dadurch gekennzeichnet, daß zwischen den Filtern (3) der Kolonne (1) und der Einrichtung (14, 30) zum Abführen der konzentrierten Suspension eine Waschzone mit einer Zufuhrpumpe (29) für Waschflüssigkeit vorgesehen ist.

8. Vorrichtung nach Anspruch 7, dadurch gekennzeichnet, daß die Einrichtung (14, 30) zum Abführen der konzentrierten Suspension in eine Spülkammer (27) debouchiert und daß Einrichtungen zum Re-Suspendieren der konzentrierten Suspension vorgesehen sind.

9. Vorrichtung nach einem oder mehreren der vorangehenden Ansprüche, dadurch gekennzeichnet, daß eine Einrichtung (29, 32) für eine kontrollierte Dosierung der Waschflüssigkeit vorgesehen ist.

10. Vorrichtung nach einem oder mehreren der Ansprüche 7—9, dadurch gekennzeichnet, daß in der Waschzone eine Rühreinrichtung (23, 24, 25) vorgesehen ist, die einen Rotor (23, 24) aufweist, dessen Achse (23) coaxial zur Mittellinie der Waschzone ist, wobei der Rotor (23, 24) wenigstens an einem Teil seiner Länge mit radial nach außen vorstehenden Stiften (24) versehen ist, und daß die Waschzone von einem zylindrischen Mantel umgeben ist, der mit radial nach innen vorstehenden Stiften (25) versehen ist.

11. Vorrichtung nach einem oder mehreren der Ansprüche 7—10, gegebenenfalls in Verbindung mit einem oder mehreren der Ansprüche 1—3 und 5 und 6, dadurch gekennzeichnet, daß die Einrichtung zum Erzeugen eines pulsierenden Stromes in der Konzentrationszone einen Kolben (33) aufweist, der periodisch die Durchflußrate der zuzuführenden Waschflüssigkeit ändert.

12. Vorrichtung zum Separieren des Stoffgemisches durch Kristallisation, dadurch gekennzeichnet, daß — gesehen in Richtung der Bewegungsrichtung der Kristalle — ein Kristallisationsator (9) vor der Einrichtung (5, 15) zum Zuführen der Suspension zu einer Kolonne nach einem oder mehreren der vorangehenden Ansprüche angeordnet ist.

13. Vorrichtung zum Separieren eines Stoffgemisches durch Kristallisation, die eine Vorrichtung zum Konzentrieren einer Suspension nach einem oder mehreren der Ansprüche 7—11 sowie nach Anspruch 12 aufweist, dadurch gekennzeichnet, daß — in Richtung des Kristallflusses gesehen — nach der Einrichtung (30) für das Abführen der konzentrierten und gewaschenen Kristallsuspension eine Einrichtung (28, 29,

32) angeordnet ist, die die abgezogene, konzentrierte und gewaschene Kristallsuspension vollständig schmilzt und anschließend teilweise als Waschflüssigkeit in die Waschzone zurückführt.

14. Kaskade zum Abscheiden von Kristallen aus einer Schmelze aufweisenden Suspension, dadurch gekennzeichnet, daß die Kaskade wenigstens zwei Vorrichtungen (101, 201) nach einem der Ansprüche 1—11 oder Anspruch 13 aufweist, die in Serie geschaltet sind, wobei die erste Vorrichtung der Serienschaltung auch gemäß Anspruch 12 ausgebildet ist, daß die Zuführungsseite der Kolonne (101) der ersten Vorrichtung mit einer Zuführungsleitung (141) für die Suspension versehen ist, daß der Auslaß (142) der Filter (103) der ersten Vorrichtung (101) mit einer Rückführungsleitung (144) für Mutterlauge versehen ist, daß der Auslaß (240) der Spülkammer (127) der ersten Vorrichtung (101) mit der Zuführungsseite der Konzentrationszone der zweiten Vorrichtung (201) verbunden ist, daß der Auslaß der Filter (203) in der zweiten Vorrichtung (201) mit der Zuführung (242) der Spülkammer (127) der ersten Vorrichtung (101) verbunden ist, und daß die Spülkammer (227) der zweiten Vorrichtung (201) über einen Wärmeaustauscher (228) mit einer Rückführungsleitung zu der Spülkammer (227) der zweiten Vorrichtung (201), der Spülkammer (127) der ersten Vorrichtung (101) und einer Produktauslaßleitung jeweils verbunden ist.

15. Kaskade zum Abscheiden von Kristallen aus einer Suspension, die eine Schmelze aufweist, nach Anspruch 14, dadurch gekennzeichnet, daß die Leitung (242) zwischen dem Auslaß (202) der Filter (203) in der zweiten Vorrichtung (201) und der Zuführung zur Spülkammer (127) der ersten Vorrichtung (101) mit einer Absperreinrichtung (148) versehen ist.

16. Kaskade zum Abscheiden von Kristallen aus einer Suspension, die eine Schmelze aufweist, nach Anspruch 15, dadurch gekennzeichnet, daß die Spülkammer (127) der ersten Vorrichtung (101) mit einer Zuführung (149) für frisches Aufgabegut versehen ist, und daß die Absperreinrichtung (148) durch die Temperatur in der ersten Spülkammer (127) gesteuert wird (145).

17. Kaskade zum Abscheiden von Kristallen aus einer Suspension, die eine Schmelze aufweist, nach den Ansprüchen 14, 15 oder 16, dadurch gekennzeichnet, daß die Kaskade aus mehr als zwei in Serie geschaltete Vorrichtungen besteht.

Revendications

1. Appareil pour concentrer une suspension de cristaux, comportant une colonne cylindrique creuse (1) qui limite une zone de concentration, la section intérieure de la colonne (1) perpendiculaire à l'axe étant constante, colonne (1) qui est munie, à une extrémité, de moyens pour l'alimentation de la suspension (5, 15), d'un ou plusieurs filtres (3) près de l'autre extrémité de

la colonne (1), de moyens pour l'enlèvement du liquide (6) passé à travers les filtres (3), de moyens pour l'enlèvement de la suspension concentrée (14, 30) à l'autre extrémité de la colonne (1) et de moyens pour générer un flux pulsatoire liquide (8, 33) dans la colonne (1), caractérisé en ce qu'un ou plusieurs tubes (2) de diamètre extérieur constant s'étendent depuis l'une des extrémités de la colonne (1) dans une direction parallèle à l'axe et sur toute la colonne (1); et en ce qu'au moins l'un desdits filtres (3) est monté dans la paroi de chaque tube (2), formant l'unique liaison directe entre l'intérieur du tube (2) et l'intérieur de la colonne (1).

2. Appareil selon la revendication 1, caractérisé en ce que la colonne (1) présente une section droite circulaire; et en ce que, si les tubes (2) sont en nombre supérieur à un, ils sont régulièrement distribués sur la section droite, en ne couvrant qu'une petite portion de cette section droite.

3. Appareil selon une ou plusieurs des revendications précédentes, caractérisé en ce que dans chaque tube (2), une portion de sa paroi, d'une longueur égale à un multiple du diamètre extérieur du tube, joue le rôle de filtre (3).

4. Appareil selon une ou plusieurs des revendications précédentes, caractérisé en ce que les moyens pour générer un flux pulsatoire dans la colonne (1) consiste en un piston (8) qui modifie périodiquement le débit d'enlèvement du liquide passé à travers les filtres (3).

5. Appareil selon une ou plusieurs des revendications précédentes, caractérisé en ce que des moyens (5, 7, 9) existent pour recycler une partie du liquide passé à travers des filtres (3) en direction de l'une des extrémités de la colonne (1).

6. Appareil selon une ou plusieurs des revendications précédentes, caractérisé en ce que les moyens (30) permettant d'enlever la suspension concentrée à l'autre extrémité de la colonne (1) comprennent une raclette (14).

7. Appareil selon une ou plusieurs des revendications précédentes, caractérisé en ce qu'entre les filtres (3) de la colonne (1) et les moyens prévus pour enlever la suspension concentrée (14, 30) est prévue une zone de lavage avec une pompe d'alimentation (29) en liquide de lavage.

8. Appareil selon la revendication 7, caractérisé en ce que les moyens prévus pour enlever la suspension concentrée (14, 30) débouchent dans une chambre de rinçage (27); et en ce que des moyens sont prévus pour remettre en suspension la suspension concentrée.

9. Appareil selon une ou plusieurs des revendications précédentes, caractérisé par des moyens (29, 32) pour commander le dosage du liquide de lavage.

10. Appareil selon une ou plusieurs des revendications 7—9, caractérisé en ce que dans la zone de lavage est prévu un moyen d'agitation (23, 24, 25) constitué d'un rotor (23, 24) dont l'axe (23) est coaxial avec l'axe de la zone de lavage, ledit rotor (23, 24) comprenant, au moins sur une partie de sa longueur, des pales (24) en saillie radialement

vers l'extérieur; et en ce que la zone de lavage est entourée par une enveloppe cylindrique pourvue de pales (25) en saillie radialement vers l'intérieur.

11. Appareil selon une ou plusieurs des revendications 7—10, éventuellement en combinaison avec une ou plusieurs des revendications 1—3 et 5 et 6, caractérisé en ce que les moyens permettant de générer un flux pulsatoire dans la zone de concentration comportent un piston (33) qui modifie périodiquement le débit d'alimentation du liquide de lavage.

12. Appareil pour séparer un mélange de substances par cristallisation, caractérisé en ce que — vu dans la direction dans laquelle se déplacent les cristaux — un cristalliseur (19) est placé en avant des moyens (5, 15) permettant d'envoyer la suspension dans une colonne conforme à une ou plusieurs des revendications précédentes.

13. Appareil pour séparer un mélange de substances par cristallisation, consistant en un appareil pour concentrer une suspension conformément à une ou plusieurs des revendications 7—11 et selon la revendication 12, caractérisé en ce que — vu dans la direction de l'écoulement des cristaux — après les moyens (30) prévus pour enlever la suspension concentrée et lavée de cristaux, est placé un dispositif (28, 29, 32) prévu pour fondre complètement, puis recycler partiellement, sous forme de liquide de lavage envoyé dans la zone de lavage, la suspension concentrée et lavée de cristaux enlevée.

14. Cascade pour séparer des cristaux à partir d'une suspension comprenant une masse fondue, caractérisée en ce que la cascade est constituée d'au moins deux appareils (101, 201), selon l'une quelconque des revendications 1—11 ou selon la revendication 13, appareils qui sont connectés en série, le premier appareil de la série étant également conforme à la revendication 12; en ce que le côté alimentation de la colonne (101) du premier appareil comporte une ligne d'alimentation (141) pour la suspension; en ce que la sortie (142) des filtres (103) du premier appareil (101) comporte une ligne de recyclage (144) pour la liqueur-mère; en ce que la sortie (240) de la chambre de rinçage (127) du premier appareil (101) est reliée au côté alimentation de la zone de concentration du second appareil (201); en ce que la sortie des filtres (203) dans le second appareil (101) est reliée à l'alimentation (242) de la chambre de rinçage (127) du premier appareil (101); et en ce que la chambre de rinçage (227) du second appareil (201) est reliée, par l'intermédiaire d'un échangeur de chaleur (228), à une ligne de recyclage en direction de la chambre de rinçage (227) du second appareil (201), à la chambre de rinçage (127) du premier appareil (101) et à une ligne d'évacuation du produit, respectivement.

15. Cascade pour séparer les cristaux à partir d'une suspension comprenant une masse fondue, selon la revendication 14, caractérisée en ce que la conduite (242) entre la sortie (202) du filtre (203) dans le second appareil (201) et l'alimentation de la chambre de rinçage (127) du premier appareil

(101) comporte un robinet-vanne de prélèvement (148).

16. Cascade pour séparer des cristaux à partir d'une suspension comprenant une masse fondue, selon la revendication 15, caractérisée en ce que la chambre de rinçage (127) du premier appareil (101) comporte une alimentation (149) en produit frais; et en ce que le robinet-vanne de prélève-

ment (148) est commandé (145) par la température qui règne dans la première chambre de rinçage (127).

17. Cascade pour séparer des cristaux à partir d'une suspension comprenant une masse fondue, selon les revendications 14, 15 ou 16, caractérisée en ce que la cascade est constituée de plus de deux appareils connectés en série.

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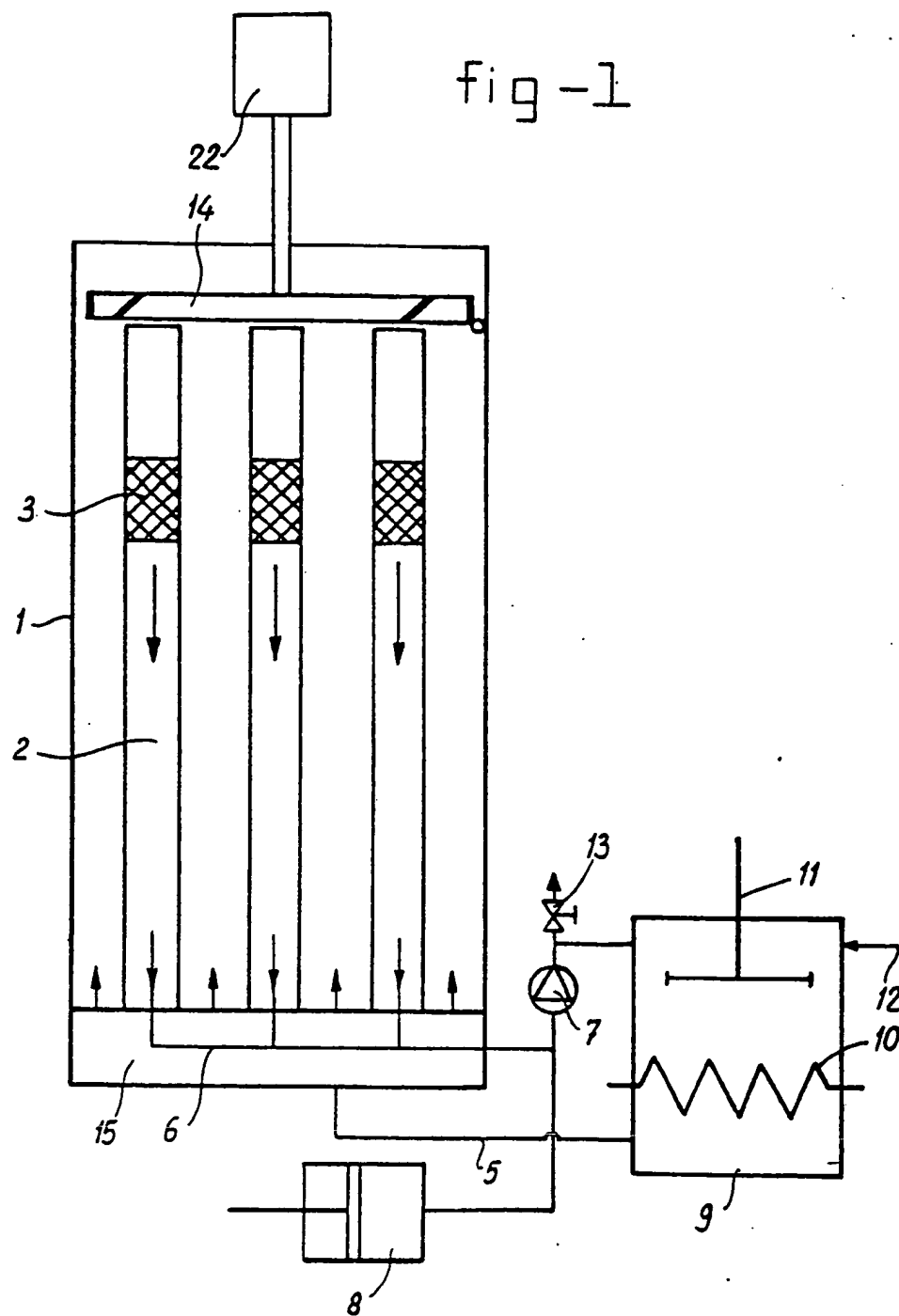
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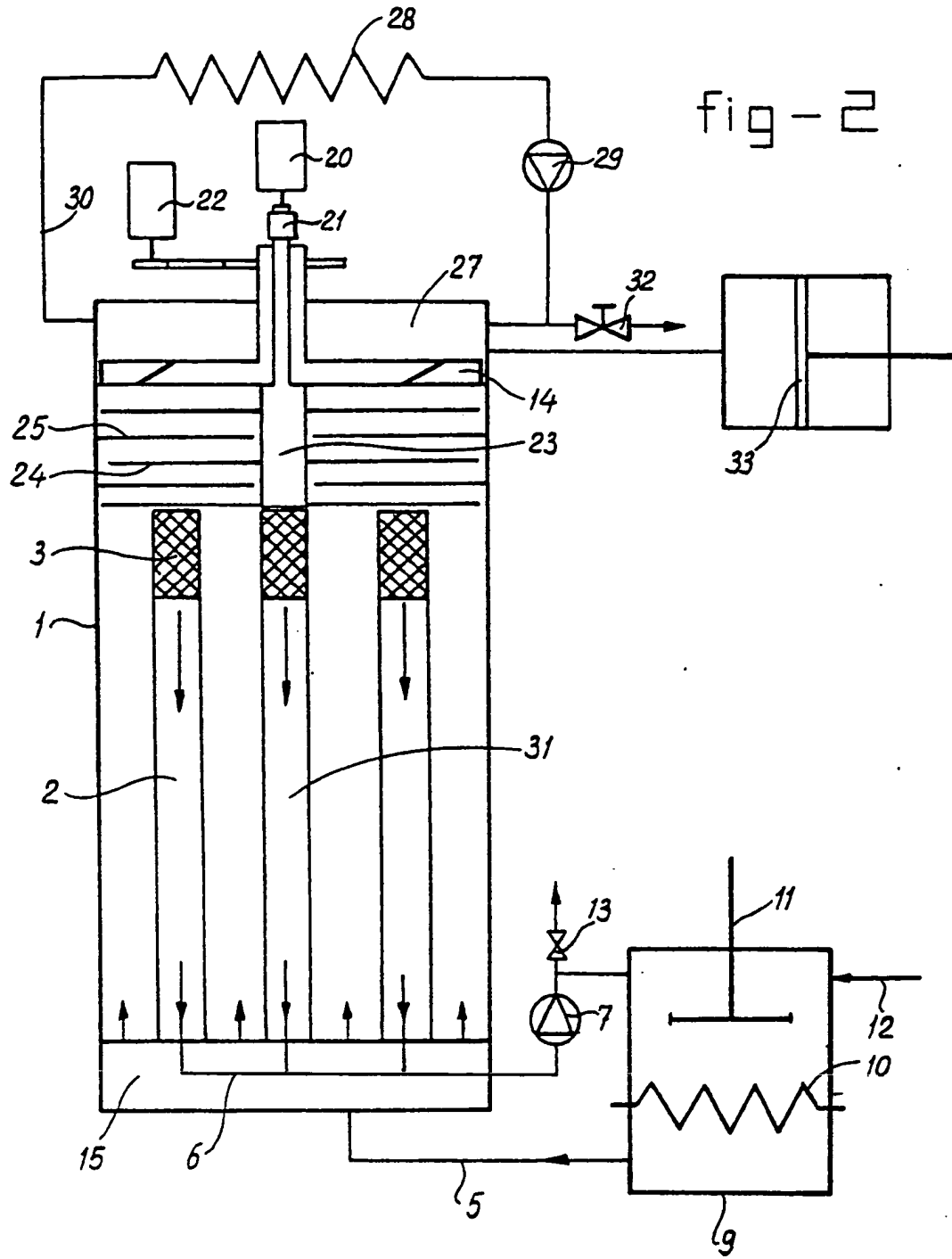
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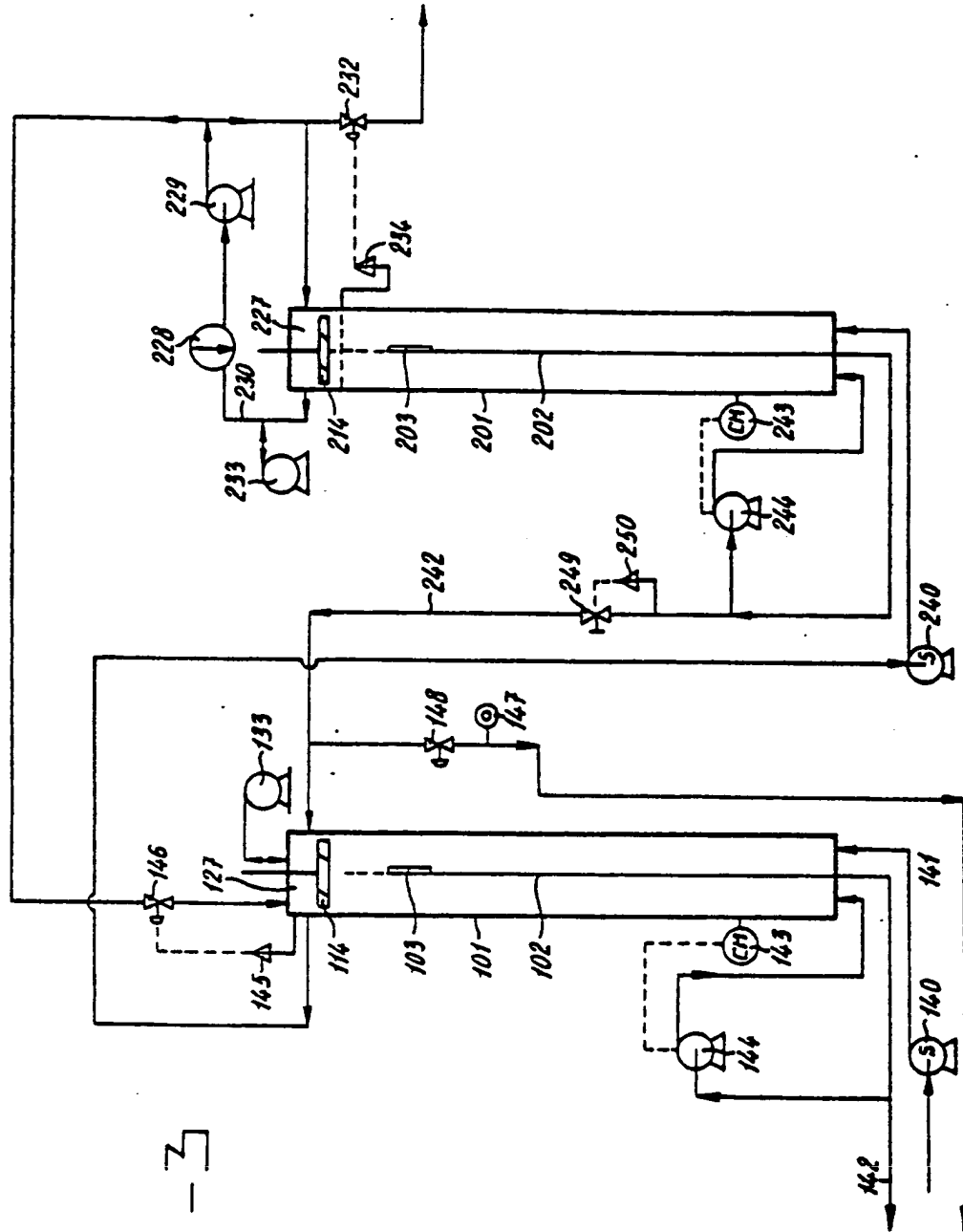


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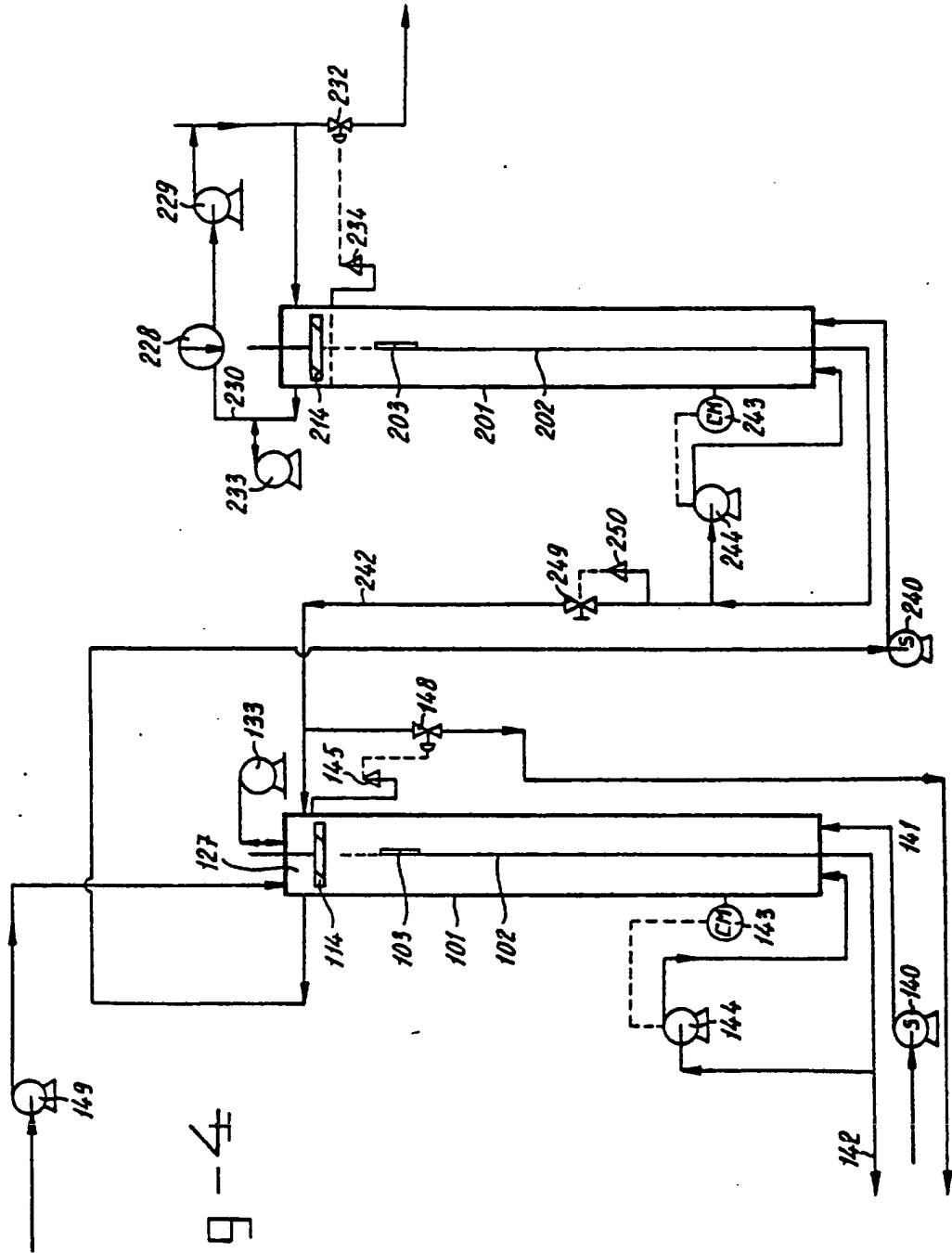


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